

# Thermal stability of hafnium bronze subjected to dynamic channel angular pressing

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**Abstract.** Thermal stability of the structure of Cu-0.8%Hf alloy subjected to severe plastic deformation by 2 passes of dynamic channel angular pressing (DCAP) has been investigated. It is demonstrated that after such deformation the microstructure of the hafnium bronze is non-uniform, where areas of cell structure are neighboring with thin-lamellar structure of twinning type. Such structure indicates the action of two deformation mechanisms, dislocation sliding and twinning. It has been revealed that doping with hafnium has considerably affected susceptibility of copper to relaxation processes under the DCAP and following annealing. It has been found that the microstructure formed under the deformation can be retained at the annealing up to 500°C without noticeable changes, and the microhardness changes only slightly in this temperature range. At the same time, at heating up to 600°C and higher the relaxation processes are developed in the structure which is accompanied with a drastic drop of microhardness.

## 1. Introduction

In the last decades materials with ultrafine-grained (UFG) structure attract great attention of the researchers due to their unique physical and mechanical properties [1, 2]. Application of severe plastic deformation (SPD) enables obtaining bulk UFG materials demonstrating unusual mechanical behavior and a complex of unique properties which are often unachievable for coarse-grained materials.

A number of various techniques of SPD have been developed by now [3]. One of the most widely used of them is the so-called equal-channel angular pressing (ECAP) [4]. At the same time, one of the new promising methods of SPD is the dynamic channel angular pressing (DCAP), which has the scheme of deformation similar to that of the ECAP, but its main difference is the high deformation rate ( $10^4 - 10^6 \text{ s}^{-1}$ ). The DCAP was worked out at the Russian Federal Nuclear Center [5]. In contrast to ECAP, for which expensive pressing equipment is required, in the DCAP the energy of powder gasses is used instead of the press, which enables to subject massive materials to SPD with high speed and high productivity. Compared to the ECAP, in case of the DCAP not only the rate of plastic deformation is increased, but also the shock-wave impact is added, which increases the SPD influence on the structure and properties of materials. Thus, application of the DCAP makes it possible to change the properties of metals and alloys substantially by the formation such structures in them



which demonstrate the combination of high strength and plasticity, as was shown in the studies of evolution of the microstructure in aluminum, copper, nickel and bronze under the DCAP [6–8]. Besides, it was found that the UFG structure can be obtained by the DCAP by the lesser numbers of passes compared to the ECAP [7, 9].

However, when various techniques of SPD are applied, relaxation processes are developed under deformation along with the microstructure refinement, which causes an attainment of the so-called saturation state, when the strain increasing does not result in further refinement and strengthening, especially in pure metals [10–12]. One of the ways to overcome this limitation is doping [8, 13, 14]. That is why it is of great interest to study the behavior of doped metals under the SPD by particularly DCAP and following heating, especially if their behavior in pure state has already been studied [6, 15]. Thus, the main goal of the present study was to investigate evolution of microstructure of hafnium bronze obtained by the DCAP under the deformation and following annealing.

## 2. Materials and methods

Specimens for investigation were made of an alloy of commercially pure copper (99.9) with 0.8 wt. % of hafnium. After casting the specimens were forged at 600°C and annealed for their homogenization at 950°C for 1 h in the vacuum with following quenching in water. This treatment resulted in the polycrystalline microstructure with an average crystallite size of about 50  $\mu\text{m}$ . The cylindrical specimens with the diameter of 10 mm and the length of 40 mm were fabricated from the as-obtained billets and subjected to DCAP at the rate of  $10^4$ – $10^5$   $\text{s}^{-1}$ . The initial speed of specimens was about 250 m/s, and the powder gases pressure was about 2 GPa. The pressing was carried out in a matrix consisting of two channels a the diameter of 10 mm intersecting under an angle of 90°, with an external rounding radius of 5 mm, which equals to a half of channels diameter, and an internal radius 1–2 mm. The DCAP was carried out in one and two passes, and after the first pass a specimen was turned by 180°. To study the thermal stability of the structure obtained the as-deformed specimens were annealed in the temperature range of 100–600°C in a high-vacuum furnace Varian for 1 h. The microstructure was studied in transmission electron microscope Tecnai G2 30 Twin. Microhardness was measured by a special unit adjusted to optical microscope Neophot-21 and calculated by the method described in detail in [14].

## 3. Results and discussion

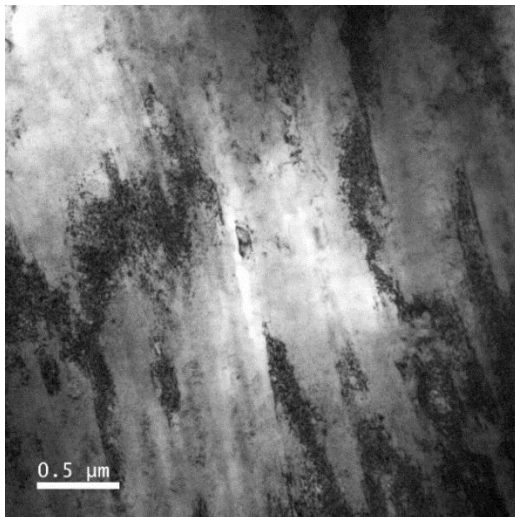
As shown by metallographic studies (Figure 1), the structure of specimens after DCAP in transverse sections is quite uniform excluding narrow areas in upper (near internal radius of matrix) and lower (near external radius) parts, which is typical of specimens subjected to the dynamic channel angular pressing [6, 8, 13, 15]. As in [6, 13], the structure in the metallographic images is mainly stringy, with curved bands resulting from the high-speed deformation by DCAP.

The more detailed information on the structure evolution in hafnium bronze and mechanisms of deformation by DCAP was obtained by transmission electron microscopy. Figure 2 demonstrates electron micrographs and diffraction patterns of the structure after two passes of DCAP and annealing at 400°C.

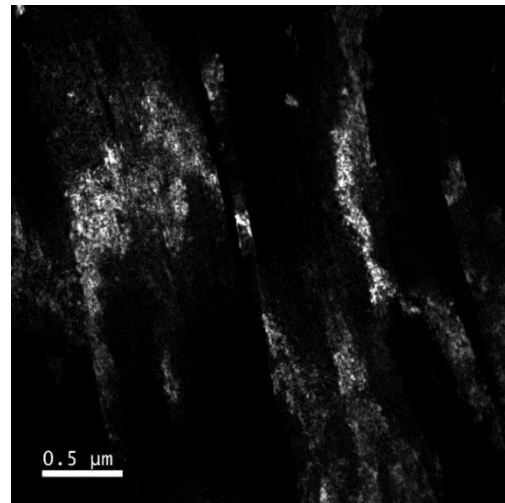
According to TEM studies, the structure after 2 passes of DCAP is non-uniform, and it is seen that there are two deformation mechanisms, dislocation sliding and twinning. These mechanisms result if the formation of two types of microstructure, namely, areas of cell structure interchanging with thin-lamellar structure of twinning type. The sizes of some cells are up to 1  $\mu\text{m}$ .



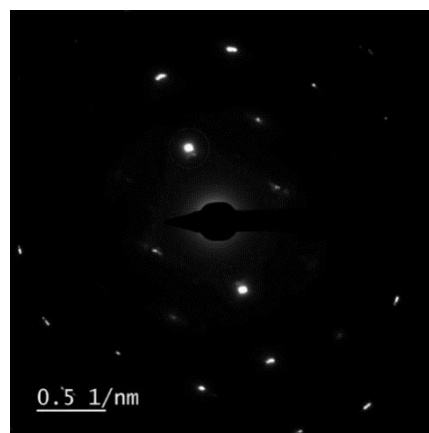
**Figure 1.** Structure of Cu-0.8Hf alloy after DCAP by 2 passes in transverse section



(a)



(b)



(c)

**Figure 2.** Microstructure of Cu-0.8Hf alloy after DCAP by 2 passes and annealing at 400°C for 1 h: (a) – bright-field image; (b) – dark-field image in (111)<sub>Cu</sub> reflection; (c) – electron diffraction pattern

Under the low-temperature annealing of the hafnium bronze the structure formed by DCAP does not undergo noticeable changes. Thus, after the annealing at 400°C the above-mentioned two types of structures – cell structure and thin-lamellar of twinning type – are present (Figure 2). The size of some cells is about 1  $\mu\text{m}$ , and they have wide dislocation boundaries. Considerable defectiveness of cells observed directly after the deformation is retained after the annealing at this temperature. The areas with twins are also retained, and micro-twins belonging to one and the same twinning system form pili-ups, and it is confirmed by the selected area diffraction patterns (Figure 2c).

It is of great importance to emphasize that the structure observed directly after the deformation by DCAP is retained without noticeable changes at annealing in the temperature range up to 400°C and even up to 500°C, thus, in the hafnium bronze under consideration the structure is more stable than, for example, in tin bronze [13]. The enhanced thermal stability is obviously due to precipitation of ultra-dispersed particles of  $\text{Cu}_5\text{Hf}$  compound [14]. However, in the electron micrographs and electron-diffraction patterns of the bronze under consideration these intermetallic precipitates are not observed, which is maybe because of their high dispersity and location in highly-defective crystallite and cell boundaries. Nevertheless, the enhanced thermal stability of the structure is confirmed by microstructure measurements. Thus, in the quenched non-deformed state the microhardness of the alloy is about  $700 \pm 20$  MPa. After the DCAP by 2 passes it increased up to 1800-2170 MPa dependently on the point of measurement, and these high values are retained at annealing up to 500°C. However, at annealing to 600°C the microhardness dramatically decreases and falls into the range of 1000-1200 MPa, which indicates the development of relaxation processes at this annealing temperature.

#### 4. Summary

According to the data obtained in this study, after the deformation by 2 passes of DCAP the structure of hafnium bronze is non-uniform, with two types of microstructure – cell structure and lamellar structure of twinning type. Such structure indicates the action of two deformation mechanisms, dislocation sliding and twinning.

It has been established, that doping of copper by hafnium considerably affects the inclination of the structure formed under the DCAP to the development of relaxation processes. It is demonstrated that the microstructure formed under the deformation is retained at annealing in the temperature range up to 500°C without noticeable changes. The microhardness changes only slightly under the annealing up to 500°C compared to its values directly after the deformation and falls in the range of 1800-2170 MPa dependently on the area of the measurements. Under the annealing at 600°C the microhardness decreases considerably to the range of 1000-1200 MPa, which indicates the development of relaxation processes.

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#### References

- [1] Gleiter H 2000 *Acta Materialia* **48** 1-29.
- [2] Valiev R, Zhilyaev A and Langdon T 2014 *Bulk Nanostructured Materials: Fundamentals and Applications* (Hoboken, New Jersey, USA: TMS, Wiley)
- [3] Valiev R, Estrin Y, Horita Z, Langdon T, Zehetbauer M and Zhu Y 2006 *The Journal of The Minerals, Metals & Materials Society* **58** (4) 33–9.
- [4] Segal V 1999 *Materials Science and Engineering: A* **271** 322–33.
- [5] Shorokhov E V, Zhgilev I N and Valiev R Z 2006 Russian Federation Patent No. 2283717
- [6] Khomskaya I, Zel'dovich V, Frolova N, Kheifets A, Shorokhov E, and Zhgilev I 2008 *The Physics of Metals and Metallography* **105** 586–93.

- [7] Popov V, Popova E, Kuznetsov D, Stolbovsky A, Shorohov E, Reglitz G, Divinski S and Wilde G 2014 *Defect Diffusion Forum* **354** 109–19.
- [8] Zel'dovich V, Frolova N, Khomskaya I, Kheifets A, Dobatkin S, Shorokhov E and Nasonov P 2016 *The Physics of Metals and Metallography* **117** 74–82.
- [9] Popov V, Popova E, Kuznetsov D, Stolbovsky A, Shorohov E, Nasonov P, Gaan K, Reglitz G, Divinski S and Wilde G 2013 *Materials Science and Engineering: A* **585** 281–91.
- [10] Pippin R, Scheriau S, Taylor A, Hafok M, Hohenwarter A and Bachmaier A 2010, *Annual Review of Materials Research* **40** 319–43.
- [11] Popova E, Popov V, Romanov E and Pilyugin V 2007 *The Physics of Metals and Metallography* **103** 407–13.
- [12] Popov V, Stolbovsky A, Popova E and Pilyugin V 2010 *Defect Diffusion Forum* **297–301** 1312–21.
- [13] Popov V, Stolbovsky A, Popova E, Falahutdinov R and Shorohov E 2017 *The Physics of Metals and Metallography* **118** 864–71.
- [14] Shangina D, Bochvar N and Dobatkin S 2016 *Inorganic Materials: Applied Research* **7** 465–70.
- [15] Khomshaya I, Zel'dovich V, Kheifets A, Shorokhov E, Frolova N, Nasonov P, Ushakov A and Zhgilev I 2011 *The Physics of Metals and Metallography* **111** 612–22.